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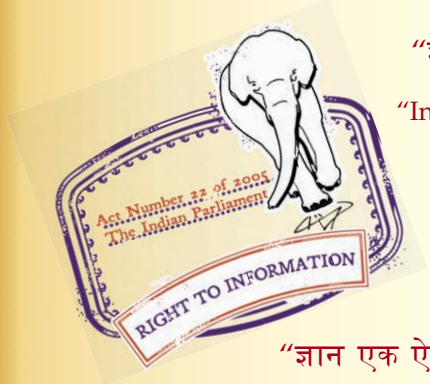

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METHOD FOR MACROETCH TEST OF WROUGHT STEEL PRODUCTS

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Indian Standard

METHOD FOR MACROETCH TEST OF WROUGHT STEEL PRODUCTS

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METHOD FOR MACROETCH TEST OF WROUGHT STEEL PRODUCTS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 5 August 1985, after the draft finalized by the Metallography and Heat Treatment Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 This method is frequently used as a evaluating technique for revealing certain aspects of the quality, structure and method of manufacture, by etching a suitably prepared specimen and examining it visually or at low magnification.

0.3 It is a widely used simple method in the steel industry, to provide information about the heterogeneity of the wrought steels. Macroetching reveals the conditions in the metal that are related to one or more of the following heterogeneities:

a) Structure, such as grain size, dendrites and columner structure;

b) Chemical composition, such as segregation, coring and banding; and

c) Presence of discontinuities, such as laps, seams, bursts, pipe and flakes.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard covers the method of macroetching, for evaluating wrought steel products, such as bars, billets, blooms, sheets, plates and forgings.

*Rules for rounding off numerical values (*revised*).

1.1.1 This method is limited in application to wrought products of carbon and alloy steels.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Dendritic Structure — Primary crystals of dendritic structure formed during solidification of steel, still remaining after working.

2.2 Ingot Pattern — A pattern developed during solidification of the ingot which appears as a zone of demarcation between the columner and heterogeneous region of ingot and persists even after reduction to the stage of inspection.

2.3 Segregation — Variation in the density of corrosive effect caused by segregation during solidification. When this phenomenon occurs at the central portion it is called centre segregation.

2.4 Porosity or Looseness — A spongy pattern caused by the development of fast corrosive effect on the entire section or the central portion of the steel material.

2.5 Pit/Pin Hole — A spotty pattern of small visible cavities caused by etching out of inclusions or microconstituents from the finished surface of the specimen.

2.6 Pipe — A discontinuity associated with segregated impurities caused by shrinkage during solidification of steel. This may be carried through the various manufacturing processes to the finished product.

2.7 Burst — A distinct pattern of cracks in the central region without any segregated impurities caused by improper forging or rolling.

2.8 Seam, Laps — A surface imperfection on wrought steel due to folding of one portion of the surface metal over another at any stage of working without being unwelded.

2.9 Thermal Cracks or Flakes — Short discontinuous internal cracks caused by stresses produced by localized transformation and hydrogen solubility effects during cooling after hot working. Also called shatter cracks and hairline cracks.

2.10 Flow Lines — Lines which appear on the polished and macroetched surface of a metal and indicate the direction in which plastic flow has taken place during fabrication.

3. SELECTION OF SAMPLE

3.1 Defects which are revealed in macroetch testing may be introduced at various stages of manufacturing. To avoid wasteful work on the defective material sampling ought to be done in an early stage of manufacturing. However, the sample should not be taken so early that further working may introduce serious defects.

3.2 A few common methods of sampling for wrought steel are indicated in **3.2.1** to **3.2.3**.

3.2.1 *For Billets, Blooms, and Hot Rolled Products* — Discs may be cut from these products near the end but not too close to the end to avoid false structure because of fish-tailing. Discs from large blooms may be cut into smaller pieces for ease of handling.

3.2.2 *For Forging and Extrusions* — Discs should be cut transverse to the long dimension to reveal flakes, bursts, etc. Forgings cut parallel to the long dimension will show flow lines. In complicated castings, to reveal flow lines, method of cutting has to be carefully selected.

3.2.3 *For Sheets and Plates* — For surface defects, a sufficiently large sample should be taken. For convenience in handling several small samples may also be taken. For lamination transverse section is taken. In case the width is too large only a portion from the centre may be taken.

4. SPECIMEN PREPARATION

4.1 If the mass of wrought steel is to be tested is relatively soft, the specimen shall be extracted by sawing or some other machining operation. In case of hard steel the specimen shall be extracted by abrasive wheel cutting. For very large section gas cutting is used but the heat affected areas shall be removed by machining or abrasive cutting wheel.

4.2 Required surface finish for specimen for macroetching vary from saw-cut machined surface to polished surfaces. The degree of surface roughness permitted depends on the severity of the etchant and also on the purpose of etching.

4.3 For satisfactory result in macroetching the smooth surface prepared by any method shall be with a minimum amount of cold work. When severe etchant is used, the discs shall be faced on lathe or shaper. The usual procedure is to take the roughing cut and then finish cut. This will prepare a smooth surface without any cold work from prior operation. Grinding shall also be conducted on the same way using free cutting wheel and light cutting wheel.

4.4 When fine details are required, a far less severe etchant is used and a smoother surface is required. The kerf marks produced by sawing operation are removed from the surface by means of filing, machine grinding or machining. Finer surface finish is obtained by grinding the specimen on No. '00' or No. '000' metallographic polishing papers.

4.5 Whatever method is used in producing smooth surface, it is important that during the operation the specimen be kept sufficiently cool to prevent heating of the surface to an excessively high temperature.

4.6 Guideline regarding surface finish required for different etching procedures has been indicated in Table 1.

4.7 After surface preparation, the sample is cleaned carefully with suitable solvents. Any greese, oil, or other residue will produce uneven attack. Once cleaned, care should be taken not to touch the sample surface or contaminate it in any way.

5. ETCHING REAGENTS

5.1 The commonly used etching reagents for wrought steels are listed in Table 1. Any other standard reagents may also be used.

6. PROCEDURE

6.1 Macroetching should be carried out in containers which shall be fairly resistant to the attack of the etching reagents. Dishes or trays made of porcelain, heat resistant glass or a corrosion resistant glass or a corrosion resistant alloy may be used as etch tanks.

6.2 The prepared specimen should be put directly into the etching solution with the sarfaces to be examined either face up or vertical to permit the gas generated to escape freely. The specimens being etched should not be too close to each other or to the tank, if it is metallic, to avoid non-uniform etching.

6.3 When etching is carried out above room temperature, the etchant should be first heated to the required temperature and then the specimen is immersed in it. To get best reproducible results, specially when the total volume of the specimen is higher to the volume of the solution, the specimen should also be heated in a water-bath, to the etching solution temperature, before immersed in the hot etchant.

6.4 The etching periods, recommended in Table 1 are only intended as a guide. The time required to develop the desired results in a particular test may be determined by frequent examination of the specimen as etching proceeds, since it depends on many factors including method of manufacture to the steel, heat treatment, alloy content, surface preparation, etc. The actual time to develop a proper structure may be quite different from the one suggested in Table 1.

6.5 Ranges of etching temperature for various etchants have been indicated in Table 1.

6.6 After completion of etching, the specimen should be washed immediately under running water using a stiff fibre brush to remove deposit of smut from the surface, rinsed again, dried with alcohol and cleaned air, and kept in a dry place.

7. INTERPRETATION OF RESULTS

7.1 Indications that may be commonly seen after macroetching are given below and also illustrated in Table 1.

7.1.1 *Centre Defects* — Pipe, bursts, segregation and porosity or looseness.

7.1.2 *Surface and Sub-surface Defects* — Seams, laps, etc; ingot corner segregation or cracks and pin holes.

7.1.3 *Miscellaneous Defects* — Thermal cracks or flakes, foreign metal foreign inclusion or 'dirt' and ingot pattern.

7.1.4 Flow line indicating direction of plastic flow.

7.1.5 Grain size.

7.2 The illustrations are examples of various indications and are not to be used as standards of acceptance or rejection.

8. REPORTING OF RESULTS

8.1 Reports should include full information on type and composition of the steel, cross-sectional dimension of the spicemen and conditions or defects observed. The observed defects may be grouped by type and location.

9. INSPECTION

9.1 Wherever macroetch testing is stipulated, agreement should be reached between the manufacturer and the purchaser regarding the following:

a) The stage of manufacture at which test shall be conducted.

b) The number and locations of the specimens to be examined.

c) The necessary surface preparation prior to etching of the specimen.

d) The etching procedure.

e) Permissible degree to which each of the defects listed in **7.1.1**, **7.1.2** and **7.1.3** may be tolerated for each of the end products.

TABLE 1 MACROETCHANTS FOR IRON AND STEEL

(*Clauses* 5.1 *and* 7.1)

Sl No.	Composition		Alloys	Temperature	Etching Time	Surface Required	Characteristics Revealed
(1)	(2)		(3)	(4)	(5)	(6)	(7)
				°C	minutes		
1.	HCl H_2O	50 ml 50 ml	Plain and alloy steels, high speed tool steels, cutlery steels and stainless steels	70-80	15-60	A or B	Segregation, porosity, hardness penetration, cracks, inclusions, dendrites, flow lines, soft spots, structure and weld examination
2.	HCl H_2SO_4 H_2O	38 ml 12 ml 50 ml	do	do	do	B or C	do
3.	HCl H_2SO_4 H_2O	50 ml 7 ml 18 ml	do	do	do	A or B	do
4.	Ammonium copper chloride H_2O	10-35 g 100 ml	Steels other than stainless and heat resisting steels	Room temperature	Immerse undisturbed for about 5 min. Remove copper deposit by cloth. Repeat the process for 3-10 times	B	Macrostructure of steel material of comparatively large size
5.	HCl HNO_3 H_2O	50 ml 25 ml 25 ml	Stainless and high alloy steels	do	10-15	A or B	Same as Sl No. 1
6.	HCl Saturated solution of $CuSO_4$ in H_2O	50 ml 25 ml	Stainless and high temperature alloys	70-80	15-30	B or C	Same as Sl No. 1

7.	HNO_3 Water or Ethanol	5-25 per-cent rest	Plain and low alloy steels	Room temperature	5-30	C	Carburization, decarburization, hardness penetration, cracks, segregation and weld examination
8.	H_2SO_4 H_2O	10 ml 90 ml	Plain, low and high alloy steels	do	24-60	C	Inclusions, porosity, pipe, blow holes on large sections
9.	H_2SO_4 H_2O	10 ml 90 ml	do	70-80	15-60	C	do
10.	$CuCl_2$, $2H_2O$	25 g	Plain and low alloy steels	Room temperature	Until the surface appears coppery	B or C	Phosphorous rich areas and banding
	$MgCl_2$, $6\ H_2O$	20 g					
	HCl	10 ml					
	Ethanol	500 ml					
11.	$(NH_4)_2S_2O_8$ H_2O	50 g 500 ml	do	do	Swab until desired etch is obtained	C	Grain size and weld examination
12.	$CuCl_2$ $FeCl_2$ HCl Ethanel	6 g 6 g 10 ml 100 ml	Low carbon steel	do	Heat the specimen to 200°C and immerse	C	Show strain lines

A = Saw cut or machined surface. B = Average ground surface. C = Polished surface.

NOTE — All acids shall be concentrated in strength. Commercial grades ordinarily may be used instead of other grades of reagents.

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